17-13-00 Total Pages Attorney Docket No. 44522 UTILITY PATENT APPLICATION First Named Inventor or Application Identifier **TRANSMITTAL** David E. Wallick et al. EK644849888US the new nonprovisional applications under 37 CFR 1.53(b) Express Mail Label No. LOW VISCOSITY ALKYL DIPHENYL OXIDE SULFONIC ACID BLENDS Title: Assistant Commissioner for Patents U APPLICATION ELEMENTS ADDRESS TO: **Box Patent Application** Washington, DC 20231 See MPEP chapter 600 concerning utility patent application contents. Microfiche Computer Program (Appendix) 6. 1. X Fee Transmittal Form (Submit an original, and a duplicate for fee processing) Nucleotide and/or Amino Acid Sequence Submission 2. [Total Pages 24] Specification (if applicable, all necessary) (preferred arrangement set forth below) Computer Readable Copy - Descriptive title of the Invention - Cross References to Related Applications Paper Copy (identical to computer copy) - Statement Regarding Fed sponsored R & D Statement verifying identity of above copies - Reference to Microfiche Appendix - Background of the Invention ACCOMPANYING APPLICATION PARTS - Brief Summary of the Invention - Brief Description of the Drawings (if filed) - Detailed Description Assignment Papers (cover sheet & document(s)) - Claim(s) English Translation Document (if applicable) Abstract of the Disclosure Information Disclosure Copies of IDS Drawing(s) (35 USC 113) 10. [Total Sheets 4] Statement (IDS)/PTO-1449 Citations **Preliminary Amendment** Declaration and Power of Attorney [Total Pages 11. 12. X Newly executed (original or copy) Return Receipt Postcard (MPEP 503) a. (Should be specifically itemized) 1 Copy from a prior application (37 CFR 1.63(d)) Certified Copy of Priority Document(s) 13. (for continuation/divisional with Box 18 completed) (if foreign priority is claimed) [Note Box 5 below] **DELETION OF INVENTOR(S)** 14. Associate Power of Attorney Signed statement attached deleting Sealed envelope containing confidential information, 15. inventor(s) named in the prior application, which Applicants may request to be expunged from see 37 CFR 1.63(d)(2) and 1.33(b). the application file. 16. X Authorization for payment of fees and Petition for Incorporation By Reference i 12 The entire disclosure of the prior application identified in Extensions of Time. Box 18 is considered as being part of the disclosure of the accompanying application and is hereby incorporated by **1**7. Other: reference therein. 18. If a CONTINUING APPLICATION, check appropriate box and supply the requisite information: Amend the specification by inserting before the first line, the sentence: "This application is a ☐ Continuation ☐ Divisional ☐ Continuation-in-part (CIP) of prior application No: _ 19. CORRESPONDENCE ADDRESS Correspondence address below 000109 PATENT TRACEMENT O NAME **ADDRESS**

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Attorney's Case No.: 44522

Application of: David E. Wallick and Timothy J. Gallagher

For: LOW VISCOSITY ALKYL DIPHENYL OXIDE SULFONIC ACID BLENDS

No. of Drawing Sheets: 4

EXPRESS MAIL MAILING LABEL NO EK644849888US

DATE OF DEPOSIT June 30, 2000

Sir:

Enclosed for filing is the above-identified application. Please charge the estimated fee to our Deposit Account No. 04-1512. An original and 2 copies of this sheet are enclosed.

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Total Number of Claims	<u>20</u>	
Less (Basic Fee)	<u>20</u>	
B. Extra Claims	$0 \times 18.00 =$	\$ <u>0</u>
Total Number Independent Claims	<u>8</u>	
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C. Extra Independent Claims	$5 \times 78.00 =$	\$ <u>390.00</u>
D. Multiple Dependent Claims Presente	ed + \$260.00 =	\$ <u>260.00</u>
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Respectfully submitted,

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Date: June 30, 2000

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant (s): David E. Wallick et al.

Attorney Docket No.: 44522

Group Art Unit: Unknown

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For: LOW VISCOSITY ALKYL DIPHENYL OXIDE SULFONIC ACID BLENDS

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DATE OF DEPOSIT June 30, 2000

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Sir:

AUTHORIZATION FOR PAYMENT OF FEES AND PETITIONS FOR EXTENSIONS OF TIME

Pursuant to 37 CFR 1.136(a)(3), please treat any concurrent or future reply in this application which requires a petition for an extension of time under 37 CFR 1.136(a)(1) as incorporating a petition for an extension of time for the appropriate length of time. Please charge any fees required under 37 CFR 1.17 in this application to Deposit Account No. 04-1512.

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LOW VISCOSITY ALKYL DIPHENYL OXIDE SULFONIC ACID BLENDS

This application claims the benefit of U.S. Provisional Application No. 60/146,395, filed July 30, 1999.

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FIELD OF THE INVENTION

This invention is directed to surfactant materials and compositions and to methods for making concentrated intermediates with good handling properties.

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BACKGROUND OF THE INVENTION

Rheological behavior is an important consideration in a liquid. An appropriate viscosity in a liquid product enables it to either be (a) usefully consumed as received or (b) conveniently received into a conditioning system for further adjustment of the viscosity to a useful value for the application. The utility of components used in a liquid blend is also affected by viscosity; and, in this regard, highly concentrated alkyl diphenyl oxide sulfonic acid as manufactured has a relatively high liquid viscosity. DOWFAXTM surfactants (DOWFAX is a trademark of The Dow Chemical Company) are good examples of products from alkyl diphenyl oxide sulfonic acids. Highly concentrated alkyl diphenyl oxide sulfonic acids have solids concentrations from about 60% to about 95% and are denoted as High Actives Acid, or HAA, herein. While the high viscosity can be moderated to acceptable levels with dilution in some HAAs, other HAAs (e.g. DOWFAX Detergent Acid) demonstrate an apparent liquid crystal region in the 40% to 80% solids range. The liquid crystal region is characterized by very high viscosity (greater than 1,000,000 centipoise) and the material is accordingly too viscous at temperatures below 40 degrees C for convenient handling. When the material is heated to render the viscosity acceptably convenient, the material is unfortunately too hot for safe handling outside of relatively expensive blending environments optimized for safe operations at such temperatures. As noted previously, DOWFAX surfactants are good examples of products from alkyl diphenyl oxide sulfonic acids. DOWFAX surfactants have two ionic charges per molecule. Each molecule consists of a pair of sulfonate groups on a diphenyl oxide

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backbone. This double charge density is largely responsible for excellent solvating and coupling action in this molecular family. DOWFAX surfactants have excellent solubility and stability in concentrated electrolytes and are resistant to oxidative and thermal degradation. DOWFAX surfactants have hydrophobes of a linear or branched alkyl group comprised of from six to sixteen carbons, depending upon the particular surfactant. Example utility of DOWFAX surfactants is in textile dyeing, polymer emulsion processing, agricultural chemical manufacturing, and (as an additive) cleaning fluid formulating.

It has been desired for some time to be able to sell High Active Acid as a concentrated product for use in formulations prior to neutralization in order to minimize shipping and handling costs respective to the surfactant product water component; however, (a) the addition of water to HAA at room temperature has traditionally not been convenient because of the high viscosity of the HAA at room temperatures and (b) most customers for the surfactant product are not conveniently availed of a blending environment for safe handling of hot HAA. Speculated benefits, therefore, of efficiency in shipping and handling and the benefits in safety from an HAA which could be blended into water at room temperature have not been realized. What is needed is an HAA having a useful viscosity at room temperature which can be added to water. The present invention solves this problem by providing HAA formulation embodiments and methods for their formulation so that an HAA having a relatively low viscosity at room temperature is provided.

SUMMARY OF THE INVENTION

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The room temperature viscosity of an alkyl diphenyl oxide sulfonic acid blend is beneficially controlled according to the invention by admixing a fatty acid having a carboxylic chain length between 1 and 12 into the alkyl diphenyl oxide sulfonic acid blend to provide between about 5 weight percentage and about 50 weight percentage of fatty acid in the admixture.

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BRIEF DESCRIPTION OF FIGURES

Figure 1 shows the impact of various levels of octanoic acid upon the viscosity of a DOWFAX alkyl diphenyl oxide sulfonic acid surfactant blend.

Figure 2 shows the impact of various levels of octanoic acid upon the viscosity of a DOWFAX alkyl diphenyl oxide sulfonic acid surfactant blend in the high viscosity range.

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Figure 3 shows the comparative impact of acetic, valeric, octanoic, and decanoic fatty acids on the viscosity of a DOWFAX alkyl diphenyl oxide sulfonic acid surfactant blend.

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Figure 4 shows a ternary phase diagram showing significant liquid crystal phase regions for water, DOWFAX Detergent Acid, and fatty acid (acetic acid and octanoic acid).

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

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Alkyl diphenyl oxide sulfonate surfactants are a Friedel-Crafts reaction product of an olefin and diphenyl oxide using AlCl₃ as a catalyst as indicated in Formula I.

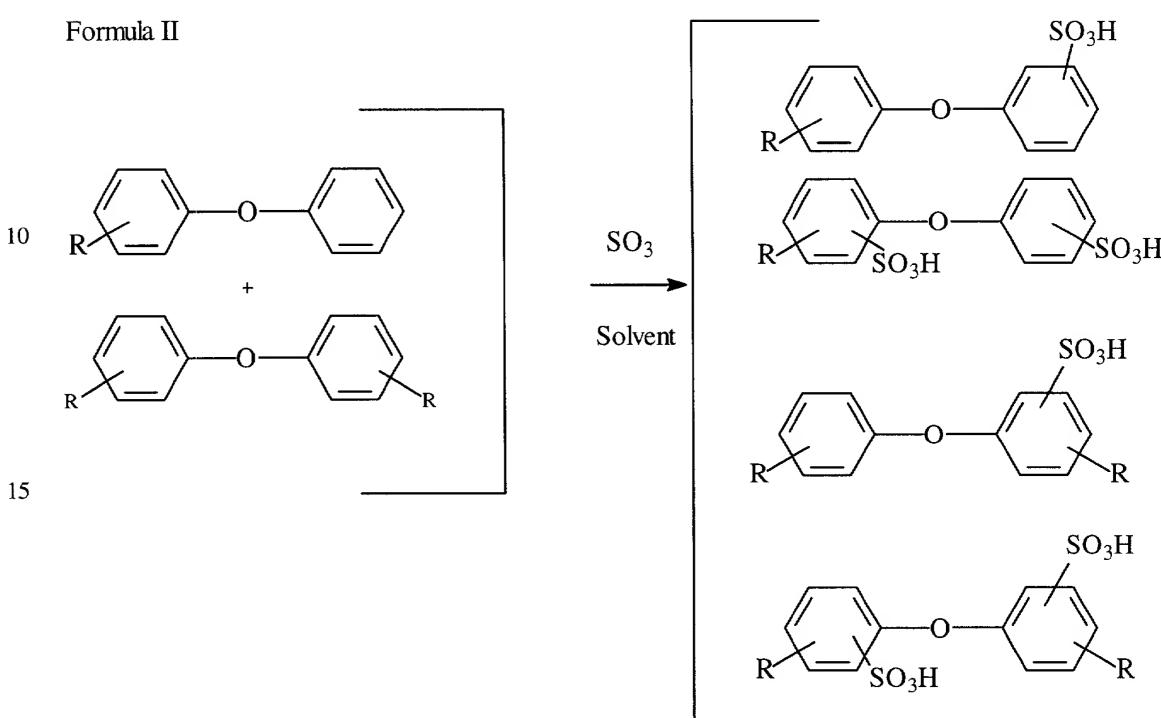
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Diphenyl oxide is present in excess and is recycled. The reaction yields a mixture of monoalkyl diphenyl oxide and dialkyl diphenyl oxide. The ratio of monoalkylation to dialkylation can be optimized depending on the end use of the products.

The next step in the process is the reaction of the alkylate with a sulfonating agent. This reaction (Formula II) is conducted in a solvent to dilute the reactant and to act as a diluent for the SO_3 used in the reaction.

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The reaction generally yields a mixture of monosulfonates and disulfonates according to Formulas III - VI. The level of disulfonation is determined by the end use of the product. Generally, the disulfonation level is above 80%. The predominant component in the commercial reaction mixture is the monoalkyl diphenyl oxide disulfonate (MADS) of Formula IV, with monoalkyl diphenyl oxide monosulfonate (MAMS) of Formula III, dialkyl diphenyl oxide monosulfonate (DAMS) of Formula V, and dialkyl diphenyl oxide disulfonate (DADS) of Formula VI essentially providing the remainder.

Formula III

$$R$$
 SO_3H SO_3H

Formula IV

$$R \xrightarrow{SO_3H} O \xrightarrow{SO_3H}$$

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Formula V
$$SO_3H$$
 R

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Formula VI
$$SO_3H$$
 R SO_3H

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Alkyl diphenyloxide sulfonates and their traditional methods of preparation are well-known and reference is made thereto for purposes of describing this invention. Representative methods of preparation and handling are disclosed in U.S. Patents 2,990,375; 3,264,242, 3,634,272; 3,945,437; and 5,015,367 which are each hereby incorporated by reference. The commercially available species are predominantly (greater than 85 percent) disulfonates (the DADS and MADS described above) and are a mixture of mono- and di- alkyl with the percentage of dialkylation (the DADS and DAMS described above) being about 5 to about 25 and the percentage of monoalkylation (the MAMS and MADS described above) being about 75 to 95 percent. Most typically,

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the commercially available species are about 85 percent monoalkyl and 15 percent dialkyl.

The traditional method taught by Steinhauer et al. (U.S. Pat. No. 2,990,375) outlines a series of steps, the first step comprising preparing an alkyldiphenyl ether by reacting an olefin or an olefin halide, such as tripropylenes, tetrapropylenes, pentapropylenes or dodecyl bromide, with diphenyl ether at a temperature between about 50° C and about 100° C in the presence of the Friedel-Crafts catalyst. The reaction mixture is washed with water to remove the catalyst, the phases separated, and the organic-rich phase subjected to distillation to obtain a fraction consisting of a mixture of monoalkylated diphenyl ether and dialkylated diphenyl ether. The number of alkyl substituents per diphenyl ether molecule can be controlled by adjusting the relative proportions of the reactants. Alternatively, the distillation can be performed so as to separate the monoalkylated and dialkylated diphenyl ethers from one another and from lower or higher boiling ingredients after which the monoalkylated and dialkylated diphenyl ether fractions can be combined at a desirable ratio.

The mixture of monoalkylated and dialkylated diphenyl ethers is subsequently reacted with a sulfonating agent, such as chlorosulfonic acid, sulfuric acid, or sulfur trioxide, in an inert solvent.

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The general process of today uses reaction of an unsaturated hydrocarbon such as an alpha-olefin in the range of 6 to 16 carbons with diphenyl oxide in the presence of AlCl₃. Reaction of alpha-olefins in the higher range of 18-30 carbons with diphenyl oxide in the presence of AlCl₃ holds some promise for fulfilling future surfactant needs. The ratio of mono- to dialkylation is controlled by the ratio of olefin to diphenyl oxide. Recycled excess diphenyl oxide is purified and reused. The rate of the reaction and the yield are controlled by the amount of catalyst and temperature of the alkylation. Excessively high temperatures as well as excessive amounts of catalyst yield higher levels of dialkylation and trialkylation. Low temperatures result in a low conversion of olefin. The ratios of concentration, catalyst and temperature are critical in keeping the reaction products consistent throughout the production cycle. The catalyst is removed from the

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process stream and the crude reaction mixture is then stripped of excess diphenyl oxide. Additional purification is optionally effected prior to the sulfonation reaction.

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Sulfonation is generally carried out in a solvent. The solvent provides value in distributing the sulfonating agent, preventing localized burning and yield loss of the reaction product, and acting as a heat removal medium in control of the reaction process temperature. Current commercial process routes use sulfur dioxide, methylene chloride, or air as reaction solvents. The air sulfonation process eliminates the need for the removal and recycle of the liquid reaction solvent and is amenable to onsite generation of SO₃. Liquid solvents require the use of liquid SO₃ that is diluted into the solvent prior to addition to the sulfonation reactors. Sulfur trioxide and chlorosulfonic acid are the two most common sulfonating agents.

After sulfonation, (1) the sulfonic acid is separated from its diluent, (2) the anhydrous acid (HAA) is diluted with water, and (3) neutralization of the diluted acid is optionally executed with an alkaline base such as sodium hydroxide. The material is packaged and sold in drums or bulk shipments as the customer requires.

The high viscosity of concentrated HAA derives from properties related to liquid crystal presence. This effect initiates at hydrophobe chain lengths above 6, is increasingly pronounced in observed samples to chain lengths of 16, and is expected to extend with greater significance to cases such as those which are contemplated via reaction of alphaolefins in the higher range of 18-30 carbons with diphenyl oxide. Accordingly, a liquid crystal disrupter, or crystal structure breaker, is highly desirable as an additive for enabling useful viscosity in a useful HAA solids region (i.e. in an 60-95% solids range). In this regard, an additional component in the blend is most desirable which disrupts High Actives Acid (HAA) liquid crystal structure without imparting undesirable attributes to the resulting blend. In this regard, dimethylformamide (DMF) and methyl formamide (MF) effectively disrupt the liquid crystal structure in alkyl diphenyl oxide sulfonic acid blends used in deriving DOWFAX surfactants; but DMF and MF are not favored for use because of asserted health concerns.

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It has been discovered that addition of fatty acids, for instance, caprylic (octanoic) or lauric acid, to highly concentrated surfactant sulfonic acid can greatly reduce the surfactant viscosity and improve handling characteristics of HAA. The use of such an additive to form particular blends enables the manufacture and use of concentrated acid forms of these surfactants.

In an alternative embodiment, admixing the fatty acid with the alkyl diphenyl oxide prior to sulfonation also provides reduction of surfactant viscosity and improved handling characteristics in the HAA material.

Formic acid, acetic acid, propionic acid, butanoic acid, pentanoic acid, valeric acid, hexanoic acid, heptanoic acid, octanoic acid, nonanoic acid, decanoic acid, undecanoic acid, and dodecanoic (lauric) acid all provide benefit in low viscosity HAA formulations as further described with reference to the sample data in the Examples and Figures.

EXAMPLE 1:

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Samples containing straight-chain carboxylic acids from formic to lauric acid were blended with a representative alkyl diphenyl oxide sulfonic acid surfactant with a 16-carbon hydrophobe side chain (DOWFAX Detergent Acid, 94 wt % concentration) at levels of 10 wt % carboxylic acid based upon DOWFAX amount. The viscosities of these samples were measured at 40 °C. The results are listed in Table 1.

Method for measuring viscosity

A Brookfield programmable rheometer, Model HDAV-III, was used to measure the viscosity of DOWFAX acid samples. The spindle size used was SC4-21. The viscosities of the samples were measured at 40 °C, a temperature at which the Thermosel temperature control stage was stable.

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Approximately 8 mLs of sample were placed into the rheometer chamber. The spindle was inserted into the chamber so that the sample covered to 1/8 inch of the spindle shaft. The chamber was placed into the temperature control stage and the spindle connected to the rheometer. The rheometer was auto-zeroed. Stirring was started at

1 RPM and the sample was allowed to temperature equilibrate for ten minutes. After the ten minutes, the motor was stopped, the sample was allowed to sit for five minutes, then the motor was started again. A reading was taken after the spindle made 5 revolutions. The stirring was increased and the torque recorded until the allowable torque range on the instrument was exceeded. The equation below was used to convert torque to viscosity in units of cP:

Viscosity = 100/RPM * TK * SMC * Torque Torque constant (TK) = 2 Spindle Multiply Constant (SMC) = 5

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TABLE 1

Structure – Viscosity Modification Attributes of Carboxylic Acid Additives in DOWFAX Detergent Surfactant

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[9.1 wt % carboxylic acid, 85.5 wt % DOWFAX Detergent, 5.4 wt % water]

10	Carboxylic Acid Common (Systematic)		Viscosity, cP (@ 40.8 °C)	
	Formic	(methanoic)	7030	
15	Acetic	(ethanoic)	5847	
	Propanoic	(propanoic)	4965	
	Butyric	(butanoic)	5227	
20	Valeric	(pentanoic)	4970	
	Caproic	(hexanoic)	6333	
25	Enanthic	(heptanoic)	6290	
	Caprylic	(octanoic)	9360	
30	Pelargonic	(nonanoic)	9120	
	Capric	(decanoic)	15820	
	Lauric	(dodecanoic)	18040	

35 EXAMPLE 2:

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Samples containing a variety of concentrations (from 2 to 50 wt % based upon DOWFAX acid amount) of a representative carboxylic acid, octanoic acid, were blended with a representative alkyl diphenyl oxide sulfonic acid surfactant with a 16-carbon hydrophobe side chain (DOWFAX Detergent Acid, or DD-HAA in Figures 1 and 2) at a variety of aqueous dilution levels (from 44 to 94 wt % DOWFAX acid). Each sample was blended until homogeneous. The viscosities of these samples were measured at 40

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°C by the method indicated in Example 1. The results of these measurements are shown in Figures 1 and 2.

Some of the samples (a) exhibited liquid crystal behavior with very high viscosities and (b) turned solid-like in consistency. These samples typically exhibited viscosities exceeding the upper measuring limit of the rheometer (1,000,000 cP), and these samples are shown as having viscosities of 1,000,000 cP in the Figures. The behavior of DOWFAX Detergent Acid containing no carboxylic acid ("0 wt % OA") is shown for comparison purposes in both Figures 1 and 2.

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The onset of the liquid crystal phase in Figure 1 is apparent at the rapid rise of viscosity with decrease of solids in the 69% to 90% solids range (depending on the particular concentration of octanoic acid). Only at 30% octanoic acid is the liquid crystal phase evidently suppressed.

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EXAMPLE 3:

Samples containing a variety of concentrations (from 2 to 30 wt %) of four representative carboxylic acids (acetic, valeric, octanoic, and decanoic acids) each were blended with a representative alkyl diphenyl oxide sulfonic acid surfactant with a 16-carbon hydrophobe side chain (DOWFAX Detergent Acid, 94 wt % concentration). Each sample was blended until homogeneous. The viscosities of these samples were measured at 40 °C by the method indicated in Example 1. The results of these measurements are shown in Figure 3. The behavior of DOWFAX Detergent Acid containing no carboxylic acid (at "0 wt % additive concentration" on the graph) is shown for comparison. Comparison of the data for all acids at concentrations above 0% in Figure 3 with the 0% case help to further illustrate the general viscosity reducing influence of fatty acids on an HAA such as the tested DOWFAX Detergent Acid.

The data of Figure 3 indicate a higher significance of fatty acid chain length toward viscosity reduction at the 5 weight percent fatty acid concentration.

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EXAMPLE 4:

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Samples containing various ratios of either acetic or octanoic acid, as representative carboxylic acids, of a representative alkyl diphenyl oxide sulfonic acid surfactant with a 16-carbon hydrophobe side chain (DOWFAX Detergent Acid), and water were prepared. Each sample was blended until homogeneous. Gross visual examination of each sample was made to identify the presence of a solid-like, liquid crystal phase. Data defining the composition of samples exhibiting such a highly viscous phase were plotted on a ternary phase diagram to ascertain the phase boundary. Boundary regions for blends with either acetic acid or octanoic acid are shown in Figure 4.

The ternary phase diagram of Figure 4 shows significant liquid crystal phase regions for water, DOWFAX surfactant acid, and two fatty acids (acetic acid and octanoic acid). The phase boundary is indicated where the viscosity measures 1 million centipoise or greater at room temperature and pressure. The high viscosity area underscores the importance of the method of addition in admixing the alkyl diphenyl oxide sulfonic acid surfactant and fatty acid blend of the described embodiments with water. It should be noted successful combination of HAA with water requires attentiveness to the issue of progression in component concentration with respect to phase control according to the depiction of Figure 4. In this regard, an alkyl diphenyl oxide sulfonic acid surfactant acid / fatty acid admixture should be added to water in use of the highly concentrated HAA in creating a surfactant for use and sale; water should not be added to the alkyl diphenyl oxide sulfonic acid surfactant acid / fatty acid admixture in use of the highly concentrated HAA in creating a surfactant for use and sale. In this regard, with reference to Figure 4, the addition of water to the alkyl diphenyl oxide sulfonic acid surfactant acid / fatty acid admixture can function to induce substantive liquid crystal formation in the admixture and render the admixture too viscous for use since the dilution of HAA with water effects entry into the liquid crystal region.

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EXAMPLE 5:

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Octanoic acid at a 10 weight percent concentration based upon expected levels of DOWFAX Detergent Acid was added to alkylate during a sulfonation reaction. A control reaction containing no octanoic acid under identical conditions yielded DOWFAX Detergent Acid exhibited a viscosity of 40,200 cP. The product of the sulfonation reaction containing the 10 weight percent octanoic acid had viscosity of 3,100 cP.

The beneficial results from use of fatty acids in the described embodiments indicate that fatty alcohols, fatty amines, or even linear alkanes in the C_6 - C_{18} range warrant consideration and empirical study in contemplated embodiment blends.

The present invention has been described in an illustrative manner. In this regard, it is evident that those skilled in the art, once given the benefit of the foregoing disclosure, may now make modifications to the specific embodiments described herein without departing from the sprit of the present invention. Such modifications are to be considered within the scope of the present invention and spirit of the appended claims.

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We claim:

- 1. A method for viscosity control in an alkyl diphenyl oxide sulfonic acid, comprising the step of:
- admixing a fatty acid having a carboxylic chain length between 1 and 12 into the alkyl diphenyl oxide sulfonic acid blend to provide between about 5 weight percentage and about 50 weight percentage of fatty acid in the admixture.
- 2. A method for preparation of an alkyl diphenyl oxide sulfonic acid blend comprising the steps of:

admixing a fatty acid selected from the group consisting of formic acid, acetic acid, propionic acid, butanoic acid, pentanoic acid, valeric acid, hexanoic acid, heptanoic acid, octanoic acid, nonanoic acid, decanoic acid, undecanoic acid, and dodecanoic acid to provide between about 5 weight percentage and about 50 weight percentage of fatty acid in the admixture with an alkyl diphenyl oxide comprising

$$R$$
 and

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where R is an alkyl radical having between 6 and 16 carbon atoms; and sulfonating said admixture with a sulfonating agent.

3. The method of Claim 2 wherein a plurality of said fatty acids are admixed in said admixing step with said alkyl diphenyl oxide blend.

- 4. A method for preparation of an alkyl diphenyl oxide sulfonic acid blend comprising the steps of:
- admixing a fatty acid selected from the group consisting of formic acid, acetic acid, propionic acid, butanoic acid, pentanoic acid, valeric acid, hexanoic acid, heptanoic acid,
- octanoic acid, nonanoic acid, decanoic acid, undecanoic acid, and dodecanoic acid to provide between about 5 weight percentage and about 50 weight percentage of fatty acid in an admixture with an alkyl diphenyl oxide sulfonic acid blend comprising

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$$SO_3H$$
 SO_3H
 SO_3H

where R is an alkyl radical having between 6 and 16 carbon atoms.

- 5. The method of Claim 4 wherein a plurality of said fatty acids are admixed admixed in said admixing step with said alkyl diphenyl oxide sulfonic acid blend.
- 6. The method of either of Claims 4 or 5 wherein the alkyl diphenyl oxide sulfonic acid blend prior to admixing of said fatty acid comprises:

between about 5 to about 25 weight percent

$$R$$
 SO_3H R and

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$$R$$
 SO_3H
 SO_3H
 R

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the constituent with the

between about 75 to about 95 respective weight percent

$$SO_3H$$
 O
and

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25 greater than 85 weight percent

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$$R$$
 SO_3H
 R
 SO_3H

- 7. The method of either of Claims 2 or 3 wherein the alkyl diphenyl oxide blend prior to admixing of said fatty acid comprises:
- between about 5 to about 25 weight percent

$$R \longrightarrow C \longrightarrow R$$
; and

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between about 75 to about 95 respective weight percent

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- 8. An alkyl diphenyl oxide sulfonic acid blend having between about 5 weight percentage and about 50 weight percentage of a fatty acid with a carboxylic chain length between 1 and 12.
- 20 9. An admixture composition of:

a fatty acid selected from the group consisting of formic acid, acetic acid, propionic acid, butanoic acid, pentanoic acid, valeric acid, hexanoic acid, heptanoic acid, octanoic acid, nonanoic acid, decanoic acid, undecanoic acid, and dodecanoic acid to provide between about 5 weight percentage and about 50 weight percentage of fatty acid in the admixture composition; and

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alkyl diphenyl oxide comprising

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$$R$$
 R R R R R R R

- where R is an alkyl radical having between 6 and 16 carbon atoms.
 - 10. The admixture composition of Claim 9 having a plurality of said fatty acids.
 - 11. An alkyl diphenyl oxide sulfonic acid blend comprising:

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a fatty acid selected from the group consisting of formic acid, acetic acid, propionic acid, butanoic acid, pentanoic acid, valeric acid, hexanoic acid, heptanoic acid, octanoic acid, nonanoic acid, decanoic acid, undecanoic acid, and dodecanoic acid to provide between about 5 weight percentage and about 50 weight percentage of fatty acid in the blend; and

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alkyl diphenyl oxide sulfonic acid comprising

$$SO_3H$$

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$$R$$
 SO_3H
 SO_3H

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$$R$$
 SO_3H R , and

$$SO_3H$$

$$SO_3H$$

$$SO_3H$$

where R is an alkyl radical having between 6 and 16 carbon atoms.

- 10 12. The blend of Claim 11 having a plurality of said fatty acids.
 - 13. The blend of either of Claims 11 or 12 wherein the alkyl diphenyl oxide sulfonic acid blend without consideration of the weight of said fatty acid comprises:

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between about 5 to about 25 weight percent

$$\begin{array}{c} SO_3H \\ \\ R \end{array} \hspace{0.5cm}, \hspace{0.5cm} \text{and} \\ \end{array}$$

$$SO_3H$$
 R
 SO_2H
 R

between about 75 to about 95 respective weight percent

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$$\begin{array}{c} SO_3H \\ \\ R \end{array} \qquad \begin{array}{c} \\ \\ \end{array} \qquad \qquad \text{and} \qquad \qquad \\ \end{array}$$

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greater than about 85 weight percent

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$$\begin{array}{c|c} & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & \\ & & & \\ & &$$

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$$\underset{R}{\underbrace{\hspace{1cm}}}_{SO_3H}$$

14. The method of either of Claims 2 or 3 wherein the alkyl diphenyl oxide blend without consideration of the weight of said fatty acid comprises:

between about 5 to about 25 weight percent

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$$R$$
 \longrightarrow R ; and

between about 75 to about 95 respective weight

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$$\mathbb{R}^{\mathbb{Z}}$$
OO

15. A method for preparation of a surfactant comprising the steps of:

admixing a fatty acid selected from the group consisting of formic acid, acetic acid, propionic acid, butanoic acid, pentanoic acid, valeric acid, hexanoic acid, heptanoic acid, octanoic acid, nonanoic acid, decanoic acid, undecanoic acid, and dodecanoic acid to provide between about 5 weight percentage and about 50 weight percentage of fatty acid in an admixture with an alkyl diphenyl oxide comprising

$$R$$
 and

where R is an alkyl radical having between 6 and 16 carbon atoms;

sulfonating said admixture with a sulfonating agent;

blending the sulfonated admixture into water; and

neutralizing the blend of water and sulfonated admixture.

16. A method for preparation of a surfactant comprising the steps of:

admixing a fatty acid selected from the group consisting of formic acid, acetic acid, propionic acid, butanoic acid, pentanoic acid, valeric acid, hexanoic acid, heptanoic acid, octanoic acid, nonanoic acid, decanoic acid, undecanoic acid, and dodecanoic acid to provide between about 5 weight percentage and about 50 weight percentage of fatty acid in an admixture with an alkyl diphenyl oxide sulfonic acid blend comprising

between about 5 to about 25 weight percent

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$$SO_3H$$

$$SO_3H$$

$$SO_3H$$

between about 75 to about 95 respective weight percent

greater than about 85 weight percent

$$\begin{array}{c|c} & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & \\ & & & \\ & &$$

$$SO_3H$$

$$R$$

$$SO_2H$$

$$R$$

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blending the sulfonated admixture into water; and neutralizing the blend of water and sulfonated admixture; where R is an alkyl radical having between 6 and 16 carbon atoms.

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ABSTRACT

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This invention addresses methods and compositions for providing alkyl diphenyl oxide sulfonic acid blends at useful viscosities for use in surfactants such as DOWFAX - containing surfactants. The low viscosity alkyl diphenyl oxide sulfonic acid blend is made by admixing a fatty acid having a carboxylic chain length between 1 and 12 (e.g., formic acid, acetic acid, propionic acid, butanoic acid, pentanoic acid, valeric acid, hexanoic acid, heptanoic acid, octanoic acid, nonanoic acid, decanoic acid, undecanoic acid, or dodecanoic acid into an alkyl diphenyl oxide sulfonic acid reaction product to provide between about 5 weight percentage and about 50 weight percentage of fatty acid in the admixture.

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VISCOSITIES OF AQUEOUS DILUTIONS OF DOWFAX DETERGENT CONTAINING VARIOUS LEVELS OF OCTANOIC ACID ADDITIVE

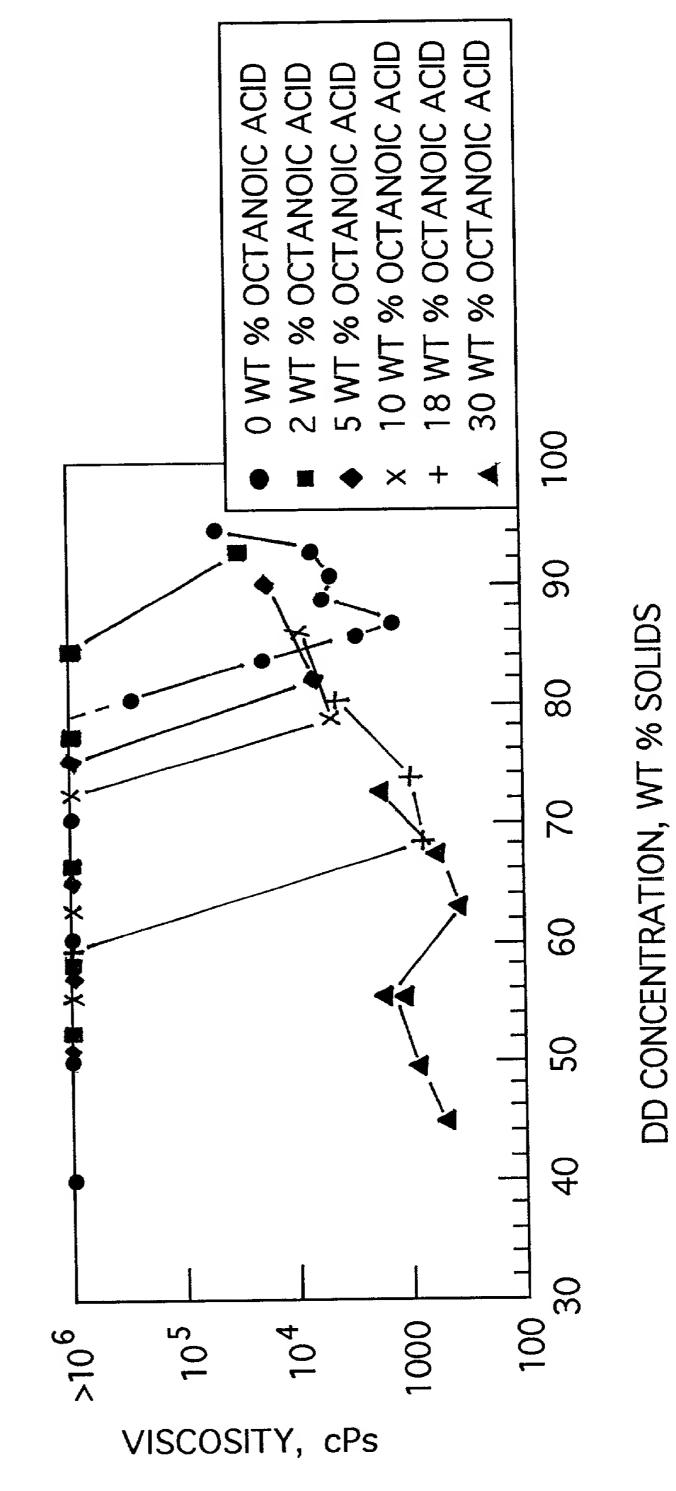
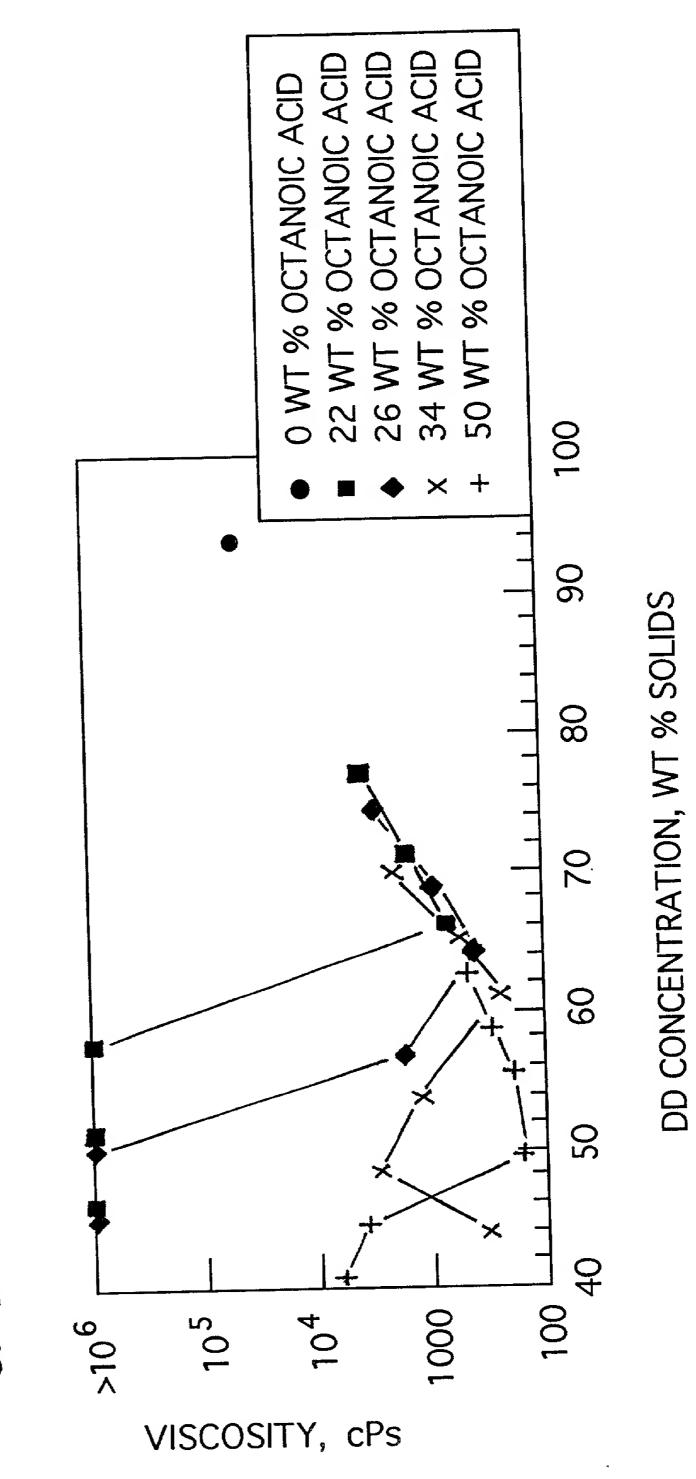


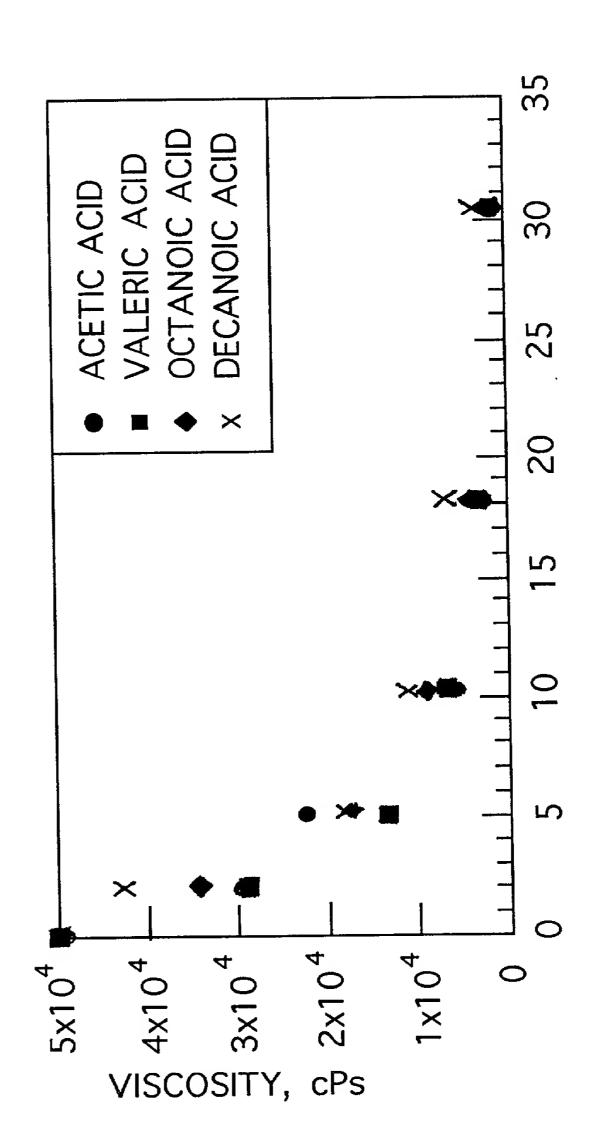
FIG. 1

CONTROL OF HIGH VISCOSITY REGION BY ADDITION OF OCTANOIC ACID TO DOWFAX DETERGENT ACID



F1G. 2

IMPACT OF ORGANIC ACID ADDITIVES ON VISCOSITY REDUCTION IN CONCENTRATED DOWFAX DETERGENT ACID



ADDITIVE CONCENTRATION, WT %

FIG. 3

